Sample Preparation: Gravity Separation Using Heavy Liquids

1 Scope

Soil samples typically contain multiple mineral species. For the purpose of identification, it is often advantageous to separate soils into their component parts. One method for accomplishing this is to separate specimens based on the specific gravity of the individual minerals. This document describes a procedure for the processing of soil samples using gravity separation in a heavy liquid by Geologist-Forensic Examiners within the Trace Evidence Unit (TEU).

2 Equipment/Materials/Reagents

- 5 ml microtube (cryogenically safe)
- Bromoform (Reagent grade)
- Ethanol (any grade)
- Sample splitter
- Containers (glass, ceramic, plastic, other appropriate material)
- Plastic container with a tight-fitting lid
- Pasteur pipette
- Gloves
- Forceps
- Laboratory coat
- Liquid nitrogen in cryogenically safe container
- Plate, flat (glass, ceramic, other appropriate material)
- Safety goggles
- Stiff paper or other appropriate material
- Additional materials may be used at the discretion of the Geologist-Forensic Examiner.

3 Standards and Controls

Not applicable.

4 Sampling or Sample Selection

4.1 Sample splitting

A split of the washed fraction of the soils is used for gravity separation. Refer to the *Geologically-Derived Materials Examinations* procedure. The sample may be split using either a microsplitter or coning and quartering.

4.1.1 If the sample is split using microsplitter, pour sample through a clean microsplitter and use one half for heavy liquid separation, retaining the second half for microscopy or other testing.

4.1.2 If sample is split using coning and quartering:

4.1.2.1 Pour washed sample onto a flat surfaced object such as a porcelain plate or a sheet of glass so that it forms a cone.

4.1.2.2 Insert a stiff yet flexible piece of paper (e.g., a business card) straight down into the center of the cone such that approximately half of the sample is on each side of the cone. Slide the card to the side, pushing half the sample aside.

4.1.2.3 Repeat step 4.1.2.2 across both piles with the card at right angles to the original split. The sample is now in four piles.

4.1.2.4 Take one quarter of the sample (one of the piles) and return it to the sample container.

4.1.2.5 Take the opposite quarter of the sample and return it to the sample container also. This material is one split.

4.1.2.6 Take the two remaining quarters and place them into another sample container. This material is the second split.

4.1.2.7 Use one of the splits for heavy liquid separation, retaining the second half for microscopy or other testing.

5 Procedure

5.1 Take one split of the sample and place it into a cryogenically safe 5 ml microtube.

5.2 Place approximately 1 ml of bromoform into the microtube and cap the microtube.

5.3 Gently swirl the sample in the bromoform.

5.4 Using a Pasteur pipette, wash down the sides of the microtube with bromoform, and recap the microtube.

5.5 Once the sample in bromoform has settled (approximately 5 minutes), immerse the bottom portion of the microtube in liquid nitrogen to slightly above the level of the heavy fraction until the liquid nitrogen stops boiling vigorously, approximately 5 seconds. The top portion of the bromoform contains the light fraction of the sample and should remain liquid.

5.6 Uncap the microtube and pour the liquid bromoform containing the light fraction into a container.

5.7 Wash down the sides of the tube with additional bromoform to remove any remaining light minerals and pour the liquid into the container used in step 5.8. Repeat this step as necessary to achieve separation.

5.8 Allow the bromoform containing the heavy fraction to thaw and then pour the bromoform containing the heavy minerals into a second container.

5.9 Wash down the sides of the tube with ethanol to remove any remaining heavy minerals and pour the liquid into the container used in step 5.8. Repeat this step as necessary to remove any remaining heavy minerals from the microtube.

5.10 Leave the containers with the light and heavy fractions in a fume hood until the bromoform has evaporated. Once the bromoform has evaporated, put each fraction in separate labeled sample containers, such as a small round plastic box. Cap the containers.

6 Calculations

Not applicable.

7 Measurement Uncertainty

Not applicable.

8 Limitations

8.1 Some components of a soil, such as asphalt containing materials, are soluble in bromoform. If it is necessary to identify those materials, this procedure may not be appropriate.

8.2 Some soil samples may be so small that processing them by this procedure is not advantageous.

9 Safety

9.1 Bromoform is a known carcinogen with acute oral toxicity to kidneys, the nervous system, liver, and upper respiratory tract. It is a skin and eye irritant. It is readily absorbed through skin contact and through vapor inhalation.

9.2 Bromoform must be handled in a fume hood with adequate exhaust ventilation.

9.3 Universal precautions will be used when handling bromoform, to include lab coat, goggles and chemical resistant gloves.

9.4 At atmospheric pressure, liquid nitrogen boils at -196°C or -32°F and is a cryogenic fluid which can cause rapid freezing on contact with living tissue and may lead to frostbite. Liquid nitrogen must be handled in insulated dewers and while wearing insulated gloves. Avoid all contact with skin.

9.5 As liquid nitrogen evaporates, it displaces oxygen in the atmosphere and may act as an asphyxiant. Liquid nitrogen must only be handled in a well-ventilated space.

9.6 Refer to the most current revision of the FBI Laboratory Safety Manual and appropriate MSDSs for additional information and guidance on hazards and handling.

10 References

- International Union of Pure and Applied Chemistry (IUPAC) *Compendium of Chemical Terminology, 2nd edition, "the Gold Book"*, "Coning and Quartering in Analytical Chemistry," compiled by A. D. McNaught and A. Wilkerson, Blackwell Scientific Publishing, Oxford, 1997.

- Geologic Materials Examinations, Trace Evidence Procedures Manual (current version)

- FBI Laboratory Safety Manual (current version)

Rev. #	Issue Date	History
1	02/07/2018	Updated throughout removing references to TEU where appropriate; added Geologist/Forensic Examiners to the Scope in Section 1. Section 4 Calibration section deleted and document renumbered. Added Sample Selection to new Section 4. Updated title of Section 7. Clarified wording in Section 8. Section 9.3 'should' to 'will'. References updated in Section 10 and throughout document.
2	09/01/2021	Added "Additional materials..." to Section 2 Equipment list. Added heading/Section 4.1. Updated formatting. Updated "Geologic Materials Analysis" to "Geologically-Derived Materials Analysis." throughout Changed "Mineralogy" to "Geology" in Approval Section. Changed TL in Approval Section.

Approval

Redact - Signatures on File

Trace Evidence Unit Chief Date: 08/31/2021

Geology Technical Leader Date: 08/31/2021